

CATEGORY:

CLEARED

527 Rec'd PCT/PTC 0.7 NOV 2000

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35.U.S.C. 371

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INTERNATIONAL APPLICATION NO PCT/JP99/02333 INTERNATIONAL FILING DATE 27 APRIL 1999 PRIORITY DATE CLAIMED 12 MAY 1998

TITLE OF BUILDING

FILM FOR FORMING VAPOR DEPOSITED BALLOON

APPLICANT(S) FOR DO/GO/US

Osamu Niwa, Hidekazu Biwaki and Takahiro Oka

Applicant herewith submits to the United States Designated /Elected Office (DO/EO/US) the following items and other information:

1. [x] This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.

- 2. [] This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
- 3. [x] This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(f).
- A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
 A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. [] is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. [] has been transmitted by the International Bureau.
 - c. [] is not required, as the application was filed in the United States Receiving Office (RO/US).
- 6. [x] A translation of the International Application into English (35 U.S.C. 371(c)(2)).
- 7. [] Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
- a. [] are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. [] have been transmitted by the International Bureau
- c. [] have not been made; however, the time limit for making such amendments has NOT expired.
- d. [] have not been made and will not be made.
- 8. [] A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
- 9. [x] An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
- [10. [] A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

- [14. [] An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
- 2. [x] An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
- 13. [] A FIRST preliminary amendment.
 - [] A SECOND or SUBSEQUENT preliminary amendment.
- 4. [] A substitute specification.
 - *15. [] A change of power of attorney and/or address letter.
 - 16. [x] Other items or information:

Verification of Translation

English translation (13 pages spec, 1 page claims, 1 page abstract)

PCT Request

International Search Report

First page of WO99/58216 Publication

PCT/IB/308

Recordation Cover Sheet for Assignment

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	27 APRIL			PRIORITY DATE CLAIMED		
17. [X] The following fees are submitted	ed:				S PTO USE ONLY	
Basic National Fee (37 CFR 1.492(a)(1)-(5):					
Neither international preliminary examina	ation fee (37 (CFR 1.482)				
Nor international search fee (37 CFR 1 44	15(a)(2)) paid	to LISPTO and Internet	ional Search			
International preliminary examination fee	(37 CFR 1.48	(2) not paid to LISPTO	but			
International preliminary examination fee	(37 CFR 1.4	82) not paid to USPTO	but			
International preliminary examination fee	paid to LISPT	O (37 CFR 1.482) but	all claims ded			
International preliminary examination fee	paid to USPT	O (37 CFR 1.482) and	all claims			
				\$860.00		
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months from the earliest claimed priority	date (37 C.)	F.R. 1.492)(e)).		s		
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Total Claims	6 -20=		X \$ 18.00	\$		
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Multiple dependent claim(s) (if applicabl	e)					
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. [] Please charge our Deposit Account	No. <u>02-437</u>	77 in amount of \$	to cover the abo	ve fees. A copy of the	his sheet is enclosed.	
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DESCRIPTION

FILM FOR FORMING VAPOR DEPOSITED BALLOON

TECHNICAL FIELD

The present invention relates to a film for forming a vapor deposited balloon and a method for manufacturing the same.

BACKGROUND ART

Conventionally used as a film for forming a vapor deposited balloon is a laminate film having a three10 layer structure consisting of a layer of nylon 6, an adhesive resin layer and a layer of LLDPE.

Further, Japanese Unexamined Patent Publication No. 290650/1995 proposes a laminate film having a five-layer structure consisting of a polyamide resin layer, a saponified copolymer layer of ethylene and vinyl acetate, a polyamide resin layer, an adhesive resin layer and a polyolefin resin layer.

These films, however, have the defect of curling.

An object of the present invention is to provide

20 a film for forming a vapor deposited balloon, which is
free of curling.

DISCLOSURE OF INVENTION

The present invention provides a film for forming a vapor deposited balloon, the film having a fivelayer structure consisting of a polyamide resin layer, a

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polyolefin layer, a polyamide resin layer, an adhesive resin layer and an LLDPE layer.

In the film for forming a vapor deposited balloon according to the invention, the polyamide resin layer comprises crystalline or amorphous nylon such as nylon 6 (poly- & -caprolactam), nylon 66 (polyhexamethylene adipamide), nylon 12 (a polymer of lactam of 12aminododecanoic acid), nylon 6-66 copolymer, nylon 6-12 copolymer or the like. The polyamide resin layer preferably comprises about 70-95 wt.% of crystalline nylon and about 30-5 wt.% of amorphous nylon, more preferably about 80-90 wt.% of crystalline nylon and about 20-10 wt.% of amorphous nylon. Nylon 6-66 copolymer is a copolymer of nylon 6 (poly- ϵ -caprolactam) and nylon 66 (polyhexamethylene adipamide), and preferably contains nylon 6 in a proportion of about 30 mole % or more, more preferably about 50 mole % or more, further more preferably about 70 mole % or more. The nylon copolymer preferably has a molecular weight of about 13,000 to about 33,000. The two polyamide resin layers of the film may be the same or different as long as they contain the above components. Each polyamide resin layer has a thickness of about 1-15 μ m, preferably about 3-10 μ m. Examples of amorphous nylons include copolymers or terpolymers of dicarboxylic acid (e.g., terephthalic acid and isophthalic

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acid) and diamine (e.g., hexamethylenediamine).

Any polyolefin layer that is capable of bonding the two polyamide resin layers together can be used without limitation. The polyolefin layer preferably 5 comprises polyolefin such as polyethylene or polypropylene, more preferably linear low density polyethylene (LLDPE) and/or low density polyethylene (LDPE), especially LLDPE. The polyolefin layer has a thickness of about 1-6 $\mu \, \mathrm{m}$, preferably about 1.5-4 μ m. The polyolefin layer may contain, in addition to or in place of polyolefin such as LLDPE, a polyolefin-based adhesive resin such as grafted LLDPE. The polyolefin layer preferably comprises a polyolefin-based adhesive resin.

Any adhesive resin layer that is capable of bonding the polyamide resin layer and the seal layer together may be used without limitation. A preferred adhesive resin is LLDPE treated by graft reaction. The adhesive resin layer has a thickness of about 1-6 $\mu\mathrm{m}$, preferably about 1.5-3 μ m.

The seal layer comprises LLDPE or LDPE, especially LLDPE. The seal layer has a thickness of about 2-20 μ m, preferably about 5-10 μ m.

The total thickness of the layers of the film for forming a balloon according to the invention is about 10-70 μ m, preferably about 15-35 μ m.

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Preferably, the polyamide shrinkable multilayer film of the invention is a flat film. The layers of the film are co-extruded from a T-die into a flat film and the film is subjected to simultaneous or sequential biaxial stretching. The laminate film may be formed by any method without limitation, but generally formed by extrusion casting on a chilled roll. The film thus obtained is simultaneously or sequentially stretched in biaxial directions, thereby giving a film of the invention. The stretching may be carried out in the machine direction using a roll stretching machine and in the transverse direction using a tenter stretching machine.

The stretch ratio in machine direction is about 1.2-5 and the stretch ratio in transverse direction is about 2.5-5. Stretching in machine direction is carried out at about $60-120^{\circ}\text{C}$, preferably about $70-100^{\circ}\text{C}$. Stretching in transverse direction is carried out at about $70-180^{\circ}\text{C}$, preferably about $100-160^{\circ}\text{C}$.

After stretching, heat treatment may optionally

20 be carried out. There is no limitation on the heat
treatment method, but the heat treatment is generally
carried out by a continuous process after transverse
stretching using a tenter stretching machine. The heat
treatment may be carried out with the film being

25 diminished (relaxed) in width by 20% or less, preferably

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by 3-10%, or with the film being set to the same width as when transversely stretched or set to a larger width than when transversely stretched. The heat treatment may be carried out at temperatures below 150° C, preferably at about $80-130^{\circ}$ C, optionally with the film being diminished in width by 20% or less, preferably by 3-10%, for example, using a tenter stretching machine. The film is subjected to thermal contraction in order to prevent natural contraction.

The multilayer film of the present invention is suitable for use as a film for forming a vapor deposited balloon. Stated more specifically, a vapor deposited layer of aluminium oxide, silicon dioxide or any other metal (e.g., aluminum foil) may be formed over the whole or partial surface of the outermost polyamide resin layer of the film of the invention. The metal deposited layer may be formed on any part of the surface of the outermost layer. However, it is preferable that the film be cut into strips and a metal vapor deposited layer be formed on one side of the centerline in width, with the other side being a transparent film. This is convenient because with use of such a film, one can very easily form a balloon having a transparent half side and a non-transparent metal deposited layer formed on the other half side. Moreover, by using the film, one can easily form a spherical balloon

having a transparent appearance in one hemisphere and a non-transparent metal deposited layer formed on the other hemisphere.

Preferably, the balloon film of the present

- 5 invention has the following physical properties:
 - Haze (measured according to ASTM D-1003): 1.0-7.0%, preferably 2.0-5.0%;
 - Tensile strength: MD (800-1500 kg/cm²), TD (800-1500 kg/cm²);
- 10 Tensile elongation: MD (80-180*), TD (80-180*); (tensile strength and tensile elongation both measured according to JIS K-6732);
 - Thermal shrinkage (measured in warm water at 100℃ for 30 seconds): MD (0-5.0%), TD (0-5.0%);
- 15 Puncture strength: NY surface (0.5 kg or more); LL surface (0.5 kg or more) (measured according to JIS P-8116);
 - Impact strength: NY surface (5.0 kg·cm or more);
 LL surface (5.0 kg·cm or more) (measured with a punching impact tester using a small ball);
 - Seal strength (pressure = 2kg/cm²; time = 1 second):
 120°C (1.0 kg/cm or more), 130°C (1.0 kg/cm or more),
 140°C (1.5 kg/cm or more), 150°C (1.5 kg/cm or more),
 160°C (1.5 kg/cm or more), 170°C (1.5 kg/cm or more);
- 25 · Oxygen permeability: 100 cc/m²·24h·20℃×65%RH or less

(measured according to ASTM D-3985);

- Wet tension strength: NY surface (45 dyn/cm or more) (measured according to JIS K-6768);
- Slipperiness: between NY-NY surfaces (both the coefficient of static friction and the coefficient of dynamic friction being 0.3 to 0.6);
 between LL-LL surfaces (both the coefficient of static friction and the coefficient of dynamic friction being 0.3 to 0.8) (measured according to ASTM D-1894).

The balloon of the present invention can be formed by heat sealing portions of seal layer(s) of the balloon film(s) together to form into a desired shape such as sphere, ellipse or the like in a desired size.

BEST MODE FOR CARRYING OUT THE INVENTION

 $\label{eq:theorem} \mbox{The present invention will be described below in} \\ \mbox{more detail with reference to Examples and Comparative} \\ \mbox{Examples.}$

[Curling Evaluation Method]

Measurement temperature and humidity: 20℃ and

20 50%RH

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- (1) a film, 100 mm \times 100 mm, is diagonally cut to make incisions,
- (2) the width and height of the film are measured after rolling up the film in the machine direction and in the transverse direction.

Criteria:

The larger the width is, the smaller curling property the film has (the quality is good).

The greater the height is, the smaller curling property the film has (the quality is good).

This is because a film having higher curling property is rolled up tightly, thus having a smaller width and a lower height.

Example 1

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The laminate film having a five-layer structure was formed using the following starting materials.

(1) Starting materials:

- Polyamide resin layer (hereinafter referred to as layer
 "A") = nvlon 6 (80 wt.%) and amorphous nylon (20 wt.%).
- 15 Polyolefin layer (hereinafter referred to as layer "B")
 = an LLDPE based adhesive resin
 - Adhesive resin layer (hereinafter referred to as layer
 "C") = an LLDPE based adhesive resin
 - Seal layer`(hereinafter referred to as layer "D") = LLDPE

(2) Production method

The layer components (A/B/A/C/D) were coextruded from a T-die to form a flat five-layer film on a chilled roll with cooling water being circulated. The film was then stretched three times in the machine direction using

a roll stretching machine at 80°C and stretched 3.8 times in the transverse direction using a tenter stretching machine in an atmosphere at 120°C . Subsequently, with the width of the film being reduced by about 4% using the tenter stretching machine, the film was thermally fixed in an atmosphere at 200°C . The film obtained had a thickness of A/B/A/C/D = 4/2/4/2/8 (µm).

Table 1 shows physical properties of the film. Example 2 $\,$

A laminate film having a five-layer structure consisting of A/B/A/C/D = 4/2/4/2/8 (μ m) in thickness was formed in the same manner as in Example 1 except that the following starting materials were used. Table 1 shows physical properties of the film.

15 Layer A = nylon 6 (80 wt.%) and amorphous nylon (20 wt.%)
Layer B = LLDPE based adhesive resin (50 wt.%) + LLDPE (50
wt.%)

Laver C = LLDPE based adhesive resin

Layer D = LLDPE

20 Example 3

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A laminate film having a five-layer structure consisting of A/B/A/C/D = 6/3/6/2/18 (μ m) in thickness was formed in the same manner as in Example 1 except that the following starting materials were used. Table 1 shows physical properties of the film.

Layer A = nylon 6 (80 wt.%) and amorphous nylon (20 wt.%)

Layer B = LLDPE based adhesive resin

Laver C = LLDPE based adhesive resin

Layer D = LLDPE

- 5 Physical properties of the films obtained in Examples 1-3 were determined according to the following methods:
 - · Haze: measured according to ASTM D-1003;
 - Tensile strength and tensile elongation: measured according to JIS K-6732;
 - Slipperiness (static friction/dynamic friction): measured according to ASTM D-1894;
 - · Thermal shrinkage: measured in warm water at 100° C for 30 seconds;
- 15 Puncture strength: measured according to JIS P-8116;
 - Impact strength: measured with a punching impact tester using a small ball;
 - Seal strength: measured at a pressure of 2kg/cm² for 1 second;
- 20 · Oxygen permeability: measured according to ASTM D-3985;
 - Wet tension strength: measured according to JIS K-6768;

-11-Table 1

	Example 1	Example 2	Example 3
Average thicknes	s (µm): 20.7	20.5	35.0
Haze (%):	2. 5	3. 2	6. 5
Tensile strength	(kg/cm ²)		
MD	960	8 5 4	8 1 9
TD	1 3 9 7	1 2 6 0	1 1 4 0
Tensile elongati	on (%)		
MD	1 6 0	154	150
TD	8 5	9 5	9 2
Slipperiness (st	atic friction/dynam	nic friction)	
NY-NY	0.58/0.51	0.49/0.42	0.46/0.38
LL-LL	0.62/0.51	0.50/0.42	0.40/0.36
Thermal shrinkag	je (%)		
MD	2. 3	1. 8	1.4
TD	1. 3	0.8	0.5
Puncture strengt	th (kg)		
NY surface	0.67	0.72	0.95
LL surface	0.60	0.62	0.75
Impact strength	(kg·cm)		
NY surface	8. 3	9. 0	9.8
LL surface	9.0	9.5	10.8

-12Table 1 (continued)

		Example 1	Example 2	Example 3
	Seal strength (kg/cm)			
	1 2 0℃	1. 2	1. 2	1. 9
5	1 3 0℃	2. 1	2. 3	3. 4
	1 4 0℃	2. 2	2. 2	3. 5
	150℃	2. 3	2. 3	3. 6
	160℃	2. 3	2. 3	3. 7
	170℃	2. 3	2. 2	3. 7
10	Oxygen permeability			
	(cc/m ² ·24h·20 [°] X65%RH)	4 3	4 2	4 0
	Wet tension strength (dyn/cm)		
	LL surface	3 6	3 6	3 6
	NY surface	5 0	5 0	5 0

15 Comparative Example 1

A laminate film having a three-layer structure consisting of A/C/D = 10/2/10 (μ m) in thickness was formed in the same manner as in Example 1 except that the following starting materials were used. The curing of the film obtained was measured. Table 2 shows the results.

Layer A = nylon 6 (80 wt.%) and amorphous nylon (20 wt.%)

Layer C = LLDPE based adhesive resin

Layer D = LLDPE

Test Example 1

The films obtained in Example 1 and Comparative

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Example 1 were tested for curling. Table 2 shows the results.

Table 2

		_Widtl	h (mm)	Lengtl	n (mm)
5		MD	TD	МD	T D
Ex	ample 1	8-10	7-9	10-12	15-17
Co	mp. Ex.1	5	5	5	5

Example 4

The film obtained in Example 1 was cut into

10 strips having a width of 100 cm. A metal vapor deposited
layer of aluminum was formed to a thickness of 400Å on
one half of the surface of layer (A), i.e., on one side of
the centerline in width. The obtained strips of the
balloon film were formed into a spherical shape by heat
15 sealing portions of seal layers of the strips together.
By feeding therein helium gas at 1.2 atm., a balloon
having a non-transparent metal deposited layer was formed.
The balloon thus obtained had a good design effect and
floated in the air for 7 days.

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CLAIMS

- A film for forming a vapor deposited balloon, which comprises a five-layer structure composed of a polyamide resin layer, a polyolefin layer, a polyamide resin layer, an adhesive resin layer and a seal layer.
- 2. The film for forming a vapor deposited balloon according to claim 1 wherein the polyamide resin layer is a mixed resin layer comprising 5 to 30 wt.% of an amorphous polyamide resin and 95 to 70 wt.% of an aliphatic polyamide resin and/or a semiaromatic polyamide resin.
- 3. A film for forming a vapor deposited balloon, which is produced by forming a metal deposited layer over the whole or partial surface of the outermost polyamide resin layer of the film according to claim 1.
- 4. A film for forming a vapor deposited balloon, which has a transparent appearance on one side and comprises a metal deposited layer formed on the other side.
- 5. A balloon comprising the film for forming a 20 vapor deposited balloon according to claim 1.
 - 6. A method for producing a vapor deposited balloon, which comprises heat sealing portions of the seal layer(s) of vapor deposited balloon film(s) of claim 1 together to form into a desired shape such as sphere, ellipse or the like in a desired size.

Abstract

The present invention provides a film for forming a vapor deposited balloon, which comprises a five
1 layer structure composed of a polyamide resin layer, a polyolefin layer, a polyamide resin layer, an adhesive resin layer and a seal layer.

[] original ☐ design

national stage of PCT.

And work

BAKER & BOTTS, LLP. FILE NO.: RBH A 3371

COMBINED DECLARATION AND POWER OF ATTORNEY

(Original, Design, National Stage of PCT, Divisional, Continuation or C-I-P Application)

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name; I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

isted below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:
FILM FOR FORMING VAPOR DEPOSITED BALLOON
This declaration is of the following type:

[] [] []	divisional continuation continuation-in-part (C-I-P)			
the spe	ecification of which: (complete (a), (b), or (c))			
(a) []	is attached hereto.			
(b) []	was filed on as Application Serial No. and	l was amended on	(if applicable).	
(c) [x]	was described and claimed in PCT International A	Application No.	filed on and	was amended on
/ / 6.34	nnlicahle).	PCT/JP99/	02333 April	27, 1999 🗸

Acknowledgement of Review of Papers and Duty of Candor

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of the subject matter claimed in this application in accordance with Title 37, Code of Federal Regulations § 1.56.

[] In compliance with this duty there is attached an information disclosure statement. 37 CFR 1.98.

Priority Claim

I hereby claim foreign priority benefits under Title 35, United States Code, § 119(a)-(d) of any foreign application(s) for patent or inventor's certificate or of any PCT International Application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT International Application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application on which priority is claimed

(complete (d) or (e))

- (d) [] no such applications have been filed.
- (e) [X] such applications have been filed as follows:

...

COUNTRY	APPLICATION NO.	DATE OF FILING (day, month, year)	DATE OF ISSUE (day, month, year)	PRIORITY CLAIMED UNDER 35 USC 119
Japan	1998-128348 —	12/05/1998		X YES NO []
				[] YES NO []
				[] YES NO []
LL FOREIGN AP	PLICATION(S), IF ANY, FILED MORE THAN 12	MONTHS (6 MONTHS FOR DESIGN) PRI	OR TO SAID APPLICATION	
				[] YES NO []
				[] YES NO []
				[] YES NO []

Claim for Benefit of Prior U.S. Provisional Application(s)

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below:

Provisional Application Number	Filing Date
§	1

Claim for Benefit of Earlier U.S./PCT Application(s) under 35 U.S.C. 120

(complete this part only if this is a divisional, continuation or C-I-P application)

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior application(s) in the manner provided by the first paragraph of Title 35, United States Code § 112, I acknowledge the duty to disclose information as defined in Title 37, Code of Federal Regulations, § 1.56 which occurred between the filling date of the prior application(s) and the national or PCT international filling date of this application:

(Application Serial No.) (Filing Date) (Status) (patented, pending, abandoned)

(Application Seral No.) (Filing Date) (Status) (patented, pending, abandoned)

Filing Date) (Status) (patented, pendi Power of Attorney

As a named inventor, I hereby appoint Dana M. Raymond, Reg. No. 18,540; Frederick C. Carver, Reg. No. 17,021; Francis J. Hone, Reg. No. 18,662, Joseph D. Garon, Reg. No. 20,420; Arthur S. Tenser, Reg. No. 18,539; Ronald B. Hildreth, Reg. No. 19,498; Thomas R. Nesbitt, Jr., Reg. No. 22,075; Robert Neuner, Reg. No. 24,316; Richard G. Berkley, Reg. No. 25,465; Richard S. Clark, Reg. No. 26,545; Robert Neuner, Reg. No. 24,316; Richard G. Berkley, Reg. No. 25,465; Richard S. Clark, Reg. No. 26,046; John D. Murnane, Reg. No. 29,836, Henry Tang, Reg. No. 29,705; Robert C. Scheinfeld, Reg. No. 31,300, John A. Fogarry, Jr., Reg. No. 22,348, Louis S. Sorell, Reg. No. 32,439 and Rochelle K. Seide Reg. No. 32,300 of the firm of BAKER & BOTTS, L.L.P., with offices at 30 Rockefeller Plaza, New York New York 10112, as attorneys to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section

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1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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